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(54) Sputtering target and a method for the manufacture thereof

(57) The surface roughness of a sputtering target is controlled and the amount of residual contaminants, the hydrogen content, and the thickness of a surface damage layer are reduced, in order to homogenize the thickness of a film formed on a substrate by sputtering and prevent nodule production and suppress nodule production to reduce particle production during sputtering.

A sputtering target with the surface roughness (Ra) not more than 1.0  $\mu\text{m}$ , the total amount of contaminants, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B, not more than 500 ppm, the hydrogen content of the surface not more than 50 ppm, and the thickness of a surface damage layer not more than 50  $\mu\text{m}$  is provided, which is manufactured by precision machining using a diamond turning tool, if required.

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## Description

## BACKGROUND OF THE INVENTION

## 1. Field of the Invention

The present invention relates to a sputtering target formed on a substrate by sputtering, with excellent uniformity in film thickness and low incidence of occurrence of nodules and particles, and a method for manufacturing thereof.

## 2. Description of the Related Art

In recent years, a sputtering method using a sputtering target has widely been employed for forming thin semiconductor films and the like.

This sputtering method is a method for forming thin films by making charged particles impinge against a sputtering target, expelling the particles of a substance constituting the sputtering target therefrom with the impinging force, and depositing these particles on a base material (substrate) such as wafer placed facing the target.

One of the problems of thin films formed by this sputtering is that film thickness is apt to be uneven. It has not been clearly known that this problem of uneven film thickness is attributable to the conditions of a target surface, and no specific measures to solve this problem have been available.

In addition, during film formation by sputtering as described above, protrusions of size ranging from several micrometers to several millimeters, called nodules, may be produced in the eroded area of the sputtering target. These nodules have such a problem that they are broken by the impingement of charged particles, thereby forming particles on a substrate during sputtering.

This production of particles increases as the number of nodules on the eroded area of the sputtering target increases. The prevention of nodule production is thus a significant problem for reducing the number of these problematic particles.

Under the recent situations in which LSI semiconductor devices have been highly integrated (4M bits, 16 M bits, 64 M bits, etc.) and wiring width has been reduced to 1  $\mu\text{m}$  or less, the above-mentioned production of particles from nodules is considered to be a critical problem.

Specifically, these particles deposit directly on the thin film formed on the substrate, or they deposit and accumulate on the surrounding wall or parts of the sputtering apparatus, then peel off and re-deposit on the thin film to cause serious problems such as breaking of wires and short-circuiting. Thus, this problem of particles has become a quite significant problem as electronic device circuits have become more highly integrated and finer.

Various efforts have been made to control operating conditions of sputtering and improve magnets in order to reduce these nodules. Since the cause of nodule production has not been clarified, however, targets designed to prevent nodule production have not been known well.

## SUMMARY OF THE INVENTION

An object of the present invention is to improve thickness uniformity of a thin film formed by sputtering and to prevent nodule production during sputtering a target and suppress particle production.

According to the present invention, there are provided:

1. A sputtering target characterized in that the surface roughness of a surface to be eroded of the sputtering target is 1.0  $\mu\text{m}$  or less in terms of center line average roughness (Ra).

2. A sputtering target characterized in that the surface roughness of a surface to be eroded of the sputtering target is 1.0  $\mu\text{m}$  or less in terms of center line average roughness (Ra) and a total amount of contaminants depositing on the surface to be eroded, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B, is 500 ppm or less.

3. A sputtering target according to aspect 2, characterized in that a total amount of contaminants metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B, is 300 ppm or less.

4. A sputtering target according to any one of aspects 1-3, characterized in that the hydrogen content of a surface to be eroded of the sputtering target is 50 ppm or less.

5. A sputtering target according to aspect 4, characterized in that the hydrogen content of the surface to be eroded of the sputtering target is 30 ppm or less.

6. A sputtering target characterized in that the surface roughness of a surface to be eroded of the sputtering target is 1.0  $\mu\text{m}$  or less in terms of center line average roughness (Ra) and the thickness of the surface damage layer of

the surface to be eroded is 50  $\mu\text{m}$  or less.

7. A sputtering target characterized in that the surface roughness of a surface to be eroded of the sputtering target is 0.2  $\mu\text{m}$  or less in terms of center line average roughness (Ra) and the thickness of the surface damage layer of the surface to be eroded is 15  $\mu\text{m}$  or less.

8. A sputtering target according to aspect 6 or 7, characterized in that a total amount of contaminants depositing on a surface to be eroded, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B, is 500 ppm or less.

9. A sputtering target according to aspect 8, characterized in that a total amount of contaminants depositing on a surface to be eroded, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B, is 300 ppm or less.

10. A sputtering target according to any one of aspects 6-9, characterized in that the hydrogen content of a surface to be eroded of the sputtering target is 50 ppm or less.

11. A sputtering target according to any one of aspects 6-9, characterized in that the hydrogen content of the surface to be eroded of the sputtering target is 30 ppm or less.

12. A method of manufacturing a sputtering target characterized in that finishing of the sputtering surface is conducted by precision machining using a diamond turning tool.

13. A method of manufacturing a sputtering target characterized in that finishing of the sputtering surface is conducted by precision machining using a diamond turning tool followed by polishing.

Further objects and advantages of the present invention will be apparent from the following description of the preferred embodiments of the present invention.

#### PREFERRED EMBODIMENTS

In order to achieve the above and other objects, the inventors of the present invention devoted themselves in research and obtained the following results.

The inventors recovered a sputtering target during use and examined it in detail. As a result, it was found that uniformity of film thickness was largely affected by surface conditions of the target and could be improved by controlling surface roughness and that nodules were apt to be produced on an uneven part of a surface to be eroded of the target and the number of produced nodules was reduced as the surface roughness of the surface to be eroded of the target was finer.

For this nodules, particles expelled from a sputtering target at a low angle are readily re-deposited on a convex part of the target, and when a re-deposition speed is faster than a speed with which a surface is eroded, particles are considered to grow as nodules. When the surface is largely uneven, it is considered that re-deposition is apt to occur and thus nodules readily grow, leading to production of a large number of nodules.

Then, when a sputtering target whose surface roughness had been controlled by mechanical processing, polishing, chemical etching, and the like was subjected to sputtering, the thickness of a thin film formed was more even and the number of nodules and particles were reduced. As a result of further investigation, residual materials from processing tools such as a turning tool, which remain on a target surface due to abrasion of the tools during machining; residual abrasives; and an increase in hydrogen content of the surface due to chemical etching were found to promote production of nodules.

The above-described residual tool materials and abrasives on a target surface induce micro-arcing (micro-discharging phenomenon) on an eroded surface and a part of the surface is melted and coagulated to form an uneven part, which serves as a new nodule-production site. It was also found that micro-arcing itself increased the number of particles.

The residual amounts of various processing tools and abrasives were examined and it was found that, when the amounts of these contaminants were reduced as thoroughly as possible, the production of nodules was suppressed and the number of particles was also decreased.

When a hydrogen content of a target surface is high during chemical etching, sputtering in the initial stage becomes unstable to make an eroded surface rougher and promote production of nodules.

Although the mechanism has not necessarily been clarified, it is assumed that a trace amount of hydrogen coming out from the target surface makes plasma unstable and sputtering occurs locally so that the target surface becomes rough.

As mentioned above, in the sputtering target of the present invention, the surface roughness of a surface to be eroded is set at 1.0  $\mu\text{m}$  or less in terms of center line average roughness (Ra). The reasons for making the surface roughness of a surface to be eroded finer are as follows: Evenness of film thickness is improved; nodules are produced selectively on an eroded surface only; and since, when a part to be eroded of a target surface is excessively uneven, particles expelled from a sputtering target at a low angle are apt to re-deposit on a convex part of the target to readily

produce nodules, prevention of such re-deposition suppresses production of nodules, resulting in a decrease in the number of particles.

When Ra becomes 1.0  $\mu\text{m}$  or less, the effects for improving the evenness of film thickness and for preventing nodule production appear so that the number of particles can be reduced.

In addition, as mentioned above, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B depositing on a surface to be eroded are constituents of the materials used for machining tools and abrasives and apt to remain as contaminants upon processing the target surface. The presence of these contaminants has problems of inducing micro-arc and generating an uneven part on a surface, which serves as a place for nodule production. Therefore, it is necessary to reduce these contaminants as thoroughly as possible.

According to the present invention, the amount of contaminants depositing on a surface to be eroded, such as metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B is 500 ppm or less.

If a total amount of these substances is 500 ppm or less, micro-arc on an eroded surface is suppressed and no uneven part serving as a new place of nodule production is developed, and thus nodule production can be prevented and appearance of particles can be suppressed.

Preferably, a total amount of metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B is 300 ppm or less. The effect of preventing nodule production can be remarkably improved by reducing a total amount of contaminants to 500 ppm or less, preferably 300 ppm or less, and decreasing a center line average roughness (Ra) to 1.0  $\mu\text{m}$  or less.

The amounts of contaminants are desirably controlled by analyzing a region within about 5  $\mu\text{m}$  from a surface using analytical methods such as Glow Discharge Mass Spectrometer (GDMS).

If a surface is smoothed by chemical etching, a hydrogen content of the surface increases and then a gas component suddenly appears microscopically and unevenness is apt to be produced on the surface during sputtering. In the sputtering target according to the present invention, however, a hydrogen content of a surface to be eroded can be decreased to 50 ppm or less by controlling the conditions for chemical etching and conducting dehydrogenation treatment following chemical etching to prevent nodule production and to reduce the number of particles.

The hydrogen contents of a surface can be analyzed by an analysis comparing between a surface part and a bulk part not including the surface part, etc.

A decrease of hydrogen content to 50 ppm or less enables improvement of the effect of preventing nodule production.

The effect to prevent nodule production can be further improved by decreasing the hydrogen content of a surface to be eroded to 50 ppm or less, preferably 30 ppm or less, decreasing a total amount of contaminants to 500 ppm or less, preferably 300 ppm or less, and reducing a center line average roughness (Ra) to 1.0  $\mu\text{m}$  or less.

In manufacturing a sputtering target, machining, polishing, chemical etching, etc. are generally used to smooth a surface for controlling surface roughness. Nodule production can be effectively prevented by specifying a total amount of contaminants and a hydrogen content as described above through selection and device of these processing methods and surface treatments, as well as controlling center line average roughness (Ra).

In manufacturing a sputtering target, machining and polishing are conducted as mentioned above, and if such processing is conducted very intensely, nodule production sometimes cannot be prevented even by controlling surface roughness. Although the reason is not necessarily clear, it is assumed that intense processing disturbs atomic arrangement and an angle of particles expelled upon sputtering is shifted to smaller and therefore deposition occurs more easily even with smaller surface roughness, that is, on a less uneven surface.

Therefore, it is necessary to reduce the thickness of the surface damage layer of a surface to be eroded of the sputtering target to 50  $\mu\text{m}$  or less. A term "surface damage layer" used herein can be defined as an area where residual stress generated from processing appears. The residual stress can be measured by a residual stress measuring method using X-ray.

If processing is performed so intensely that the thickness of a surface damage layer exceeds 50  $\mu\text{m}$ , no effect of reducing the number of nodules is exerted and the number of particles cannot be reduced effectively.

In addition, in the present invention, a method of manufacturing a sputtering target in which finishing of the sputtering surface is conducted by precision machining using a diamond turning tool or by precision machining using a diamond turning tool followed by polishing, if required, has been investigated.

It has been found that precision machining using a diamond turning tool can reduce surface roughness effectively without using wet polishing or chemical polishing, which have been required conventionally. In addition, by choosing this processing condition, center line average roughness (Ra) can be 0.2  $\mu\text{m}$  or less and the thickness of a surface damage layer can be 15  $\mu\text{m}$  or less.

When a conventional carbide tool is employed, contamination of heavy metals is necessarily apt to occur. On the other hand, when a diamond turning tool is employed, such contamination does not occur.

The present method does not require characteristically washing and dehydration treatment (required in chemical

polishing) which have been required for wet polishing and chemical polishing. It is needless to say that the combined use of conventional polishing methods is allowed and can further reduce surface roughness and the thickness of a surface damage layer.

Since the quality of a film exhibited dispersions immediately after initiation of use of a target, dummy sputtering is carried out until film formation is stabilized.

As to sheet resistance, for example, since a target with dispersion of standard deviation of sheet resistance in a wafer during processing of about 3% is generally used, above-mentioned dummy sputtering is carried out until dispersion of standard deviation of sheet resistance in the wafer becomes 3% or less after initiation of using the target.

This dummy sputtering has a problem. For targets, especially those in which surface processing status has not been controlled, cumulative input power of about 20 kWh is generally required, resulting in large time loss in the process requiring film formation with low power.

According to the present invention, precision machining using a diamond turning tool enables manufacturing of a sputtering target wherein the surface roughness of a surface to be eroded of the sputtering target is 0.2  $\mu\text{m}$  or less in terms of center line average roughness (Ra) and the thickness of a surface damage layer of the surface to be eroded is 15  $\mu\text{m}$  or less as described above, and a decrease in the thickness of a surface damage layer mentioned above provides an effect to largely reduce a time required for this dummy sputtering.

#### Examples and Comparative Examples

Next, the present invention will be described in detail referring to preferred embodiments and comparative examples.

(Examples 1-9 and Comparative Examples 1-2)

Sputtering target material composed of highly purified titanium was subjected to lathe machining. Then, a surface to be eroded was subjected to diamond-finishing machining, wet polishing, chemical polishing, washing with ultra-pure water, and dehydrogenation treatment to prepare a sputtering target (300 mm in diameter and 6.35 mm in thickness) with controlled surface roughness (Ra), total amount of contaminants, hydrogen content, and thickness of a surface damage layer. These examples are shown in Tables 1 and 2. In Table 1, a round mark means that a processing or treatment was conducted.

The target thus obtained was connected to a copper backing plate with a diameter of 348 mm and a thickness of 21.0 mm and then subjected to sputtering below.

[Table 1]

[Table 2]

The sputtering target thus prepared was set in a DC magnetron sputtering apparatus and sputtering was carried out under a nitrogen atmosphere to form a TiN film on a silicon wafer. The number of nodules, average particle number, and standard deviation of sheet resistance ( $\phi 8''$ ) in a wafer at the time of cumulative input power after initiation of use being 10 kWh were examined for Examples 1-9 and Comparative Examples 1-2. The results are shown in Table 3.

[Table 3]

As shown in Table 2, the center line average roughness, Ra, was 1.0  $\mu\text{m}$ , the upper limit, or less in Examples 1-9 according to the present invention, whereas the center line average roughness was 1.8  $\mu\text{m}$  for Comparative Example 1 and 3.0  $\mu\text{m}$  for Comparative Example 2, which exceed the above-mentioned upper limit.

In addition, the total amount of contaminants depositing on the surface to be eroded, that is, metal elements with a high melting point other than the major component and alloy components (W, Ta, Mo, Nb, and the like), and Si, Al, Co, Ni, and B was 500 ppm or less, that is, in a range of 40 ppm and 470 ppm for Examples 1-12, whereas the total amounts were 670 ppm and 450 ppm for Comparative Examples 1 and 2, respectively, which values exceeded the upper limit of 500 ppm or were close to it.

The hydrogen content of the surface ranged from 8 ppm to 40 ppm in Examples 1-12. The contents were 15 ppm and 10 ppm for Comparative Examples 1 and 2, respectively.

The thickness of the surface damage layer of the surface was 5-40  $\mu\text{m}$  for Examples 1-9, while it was 70  $\mu\text{m}$  for Comparative Example 1 and 30  $\mu\text{m}$  for Comparative Example 2, which exceeded the upper limit of 50  $\mu\text{m}$  or was relatively large.

As shown from a comparison between Tables 2 and 3, the number of nodules and average particle number were

small and standard deviation of sheet resistance in a wafer was small for Examples 1-9 according to the present invention, in which none of center line average roughness; total amount of contaminants depositing on the surface to be eroded, metal elements with a high melting point other than the major component and alloy components (W, Ta, Mo, Nb, and the like) and Si, Al, Co, Ni, and B; hydrogen content; or thickness of the surface damage layer of the surface, exceeded the respective upper limits, indicating that good sputtering targets were obtained.

However, in Examples 1 and 4, in which one or more of surface roughness, amount of contaminants, hydrogen content, and thickness of the surface damage layer exhibited values close to the upper limits, the number of nodules and average particle number tended to relatively increase and the standard deviation of sheet resistance in a wafer was slightly high. Consequently, it is shown that these increases affect the properties of sputtering targets.

In Examples 7-9 in which diamond-finishing machining was performed, the center line average roughness (Ra) was 0.07-0.17  $\mu\text{m}$ , the amount of contaminants was 40-100 ppm, and the thickness of the surface damage layer was 5-11  $\mu\text{m}$ , and thus the center line average roughness (Ra) and the thickness of the surface damage layer exhibited remarkably low values.

In addition, in this case, the numbers of nodules produced and average particle numbers were small and the standard deviation of sheet resistance in a wafer at the time of cumulative input power after initiation of use being 10 kWh was constantly low, 2.1-2.5%.

From above, diamond-finishing machining is found to exert a quite excellent effect.

In Examples 8 and 9, as shown in Table 1, in addition to diamond-finishing machining, wet polishing and washing or chemical polishing, washing, and dehydrogenation treatment were conducted. The number of nodules and average particle number, and the standard deviation of sheet resistance in a wafer were further improved, showing excellent results.

On the contrary, in Comparative Examples 1 and 2, as shown in Table 3, the number of nodules was 500/target, the average number of appearing particles were 110 and 87/wafer, and the standard deviations of sheet resistance were 4.8% and 3.2%, all these values were high. Comparative Example 1, in which the amount of contaminants was particularly high and the surface damage layer was thick, showed the worst results among the Comparative Examples shown in Table 3.

From above, a titanium target according to the present invention is shown to be an excellent target with lower incidences of nodule production and particle production and a small standard deviation of sheet resistance in a wafer.

(Examples 10-14 and Comparative Examples 3-4)

Next, examples of application of the present invention to tantalum (Examples 10-14) and Comparative Examples 3-4 are shown.

Sputtering target material composed of highly purified tantalum (Ta) was subjected to lathe machining. Then, a surface to be eroded was subjected to diamond-finishing machining, wet polishing, chemical polishing, washing with ultra-pure water, and dehydrogenation treatment to prepare a sputtering target (300 mm in diameter and 6.35 mm in thickness) with controlled surface roughness (Ra), total amount of contaminants, hydrogen content, and thickness of a surface damage layer. They are shown in Tables 4 and 5. In Table 4, a round mark means that a processing or treatment was conducted.

The target thus obtained was connected to a copper backing plate with a diameter of 348 mm and a thickness of 21.0 mm and then subjected to sputtering below.

[Table 4]

[Table 5]

The tantalum sputtering target thus prepared was set in a DC magnetron sputtering apparatus and sputtering was carried out under a nitrogen atmosphere to form a TaN film on a silicon wafer. The number of nodules, average particle number, and standard deviation of sheet resistance in a wafer at the time of cumulative input power after initiation of use being 10 kWh ( $\phi 8''$ ) were examined for Examples 10-14 and Comparative Examples 3-4. The results are shown in Table 6.

[Table 6]

As shown in Table 5, the center line average roughness, Ra, was 1.0  $\mu\text{m}$ , the upper limit, or less in Examples 10-14 according to the present invention, whereas the center line average roughness was 2.2  $\mu\text{m}$  for Comparative Example 3 and 3.5  $\mu\text{m}$  for Comparative Example 4, which indicated rough surface.

In addition, the total amount of contaminants depositing on the surface to be eroded, that is, metal elements with a

high melting point other than the major component and alloy components (W, Ti, Mo, Nb, and the like) and Si, Al, Co, Ni, and B, was 500 ppm or less, that is, in a range of 35 ppm and 320 ppm for Examples 10-14, whereas the total amounts were 560 ppm and 480 ppm for Comparative Examples 3 and 4, respectively, which values exceeded the upper limit of 500 ppm or were close to the upper limit.

The hydrogen content of the surface ranged from 8 ppm to 25 ppm in Examples 10-14, and 10 ppm in Comparative Examples 3 and 4.

The thickness of the surface damage layer of the surface was 8-30  $\mu\text{m}$  for Examples 10-14, while it was 55  $\mu\text{m}$  for Comparative Example 3 and 30  $\mu\text{m}$  for Comparative Example 4, which exceeded the upper limit of 50  $\mu\text{m}$  or was relatively large.

As shown from a comparison between Tables 5 and 6, the number of nodules and average particle number were small and standard deviation of sheet resistance in a wafer was small for Examples 10-14 according to the present invention, in which none of center line average roughness; total amount of contaminants depositing on the surface to be eroded, that is, metal elements with a high melting point other than the major component and alloy components (W, Ti, Mo, Nb, and the like) and Si, Al, Co, Ni, and B; hydrogen content; or thickness of the surface damage layer of the surface, exceeded the respective upper limits, indicating that good sputtering targets were obtained.

However, in Example 10, in which the amount of contaminants and the thickness of the surface damage layer exhibited slightly larger values as compared with other Examples, the number of nodules and average particle number tended to relatively increase and the standard deviation of sheet resistance in a wafer was slightly higher as in the case of the above-mentioned TiN.

Although Example 10 is within the scope of the present invention and had no particular problem, it is shown that the increases in the amount of contaminants and thickness of a surface damage layer impose influence on the properties of sputtering targets.

In Examples 12-14 in which diamond-finishing machining was performed, the center line average roughness ( $R_a$ ) was 0.05-0.14  $\mu\text{m}$ , the amount of contaminants was 35-150 ppm, and the thickness of the surface damage layer was 8-10  $\mu\text{m}$  and thus the center line average roughness ( $R_a$ ) and the thickness of the surface damage layer were remarkably reduced.

In addition, in this case, the number of nodules produced and average particle number were small and the standard deviation of sheet resistance in a wafer at the time of cumulative input power after initiation of use being 10 kWh was constantly low, 2.3-2.7%.

From above, diamond-finishing machining is found to exert a quite excellent effect as in Examples 5-7.

In Examples 13 and 14, as shown in Table 4, in addition to diamond-finishing machining, wet polishing and washing or chemical polishing, washing, and dehydrogenation treatment were conducted. The number of nodules and average particle number, and the standard deviation of sheet resistance in a wafer were further improved, showing excellent results.

On the contrary, in Comparative Examples 3 and 4, as shown in Table 6, the number of nodules was 500/target, the average numbers of appearing particles were as high as 110 and 87/wafer, and the standard deviations of sheet resistance were 4.6% and 3.3%, showing a quite bad result.

From above, a tantalum target of the present invention is shown to be an excellent target with low incidences of nodule production and appearance of particles, and a small standard deviation of sheet resistance in a wafer.

(Examples 15-20 and Comparative Examples 5-6)

Next, examples of application of the present invention to copper (Examples 15-20) and Comparative Examples 5-6 are shown.

Sputtering target material composed of highly purified copper (Cu) was subjected to lathe machining. Then, a surface to be eroded was subjected to diamond-finishing machining, wet polishing, chemical polishing, washing with ultra-pure water, and dehydrogenation treatment to prepare a sputtering target (300 mm in diameter and 6.35 mm in thickness) with controlled surface roughness ( $R_a$ ), total amount of contaminants, hydrogen content, and thickness of a surface damage layer.

These examples are shown in Tables 7 and 8. In Table 7, a round mark means that a processing or treatment was conducted.

The target thus obtained was connected to a copper backing plate with a diameter of 348 mm and a thickness of 21.0 mm and then subjected to sputtering below.

[Table 7]

[Table 8]

The sputtering target thus prepared was set in a DC magnetron sputtering apparatus and sputtering was carried out under a nitrogen atmosphere to form a Cu film on a silicon wafer. The number of nodules, average particle number, and standard deviation of sheet resistance ( $\phi 8"$ ) in a wafer at the time of cumulative input power after initiation of use being 10 kWh were examined for Examples 15-20 and Comparative Examples 5 and 6. The results are shown in Table 9.

[Table 9]

As shown in Table 8, the center line average roughness, Ra, was 1.0  $\mu\text{m}$ , the upper limit, or less in Examples 15-20 according to the present invention, whereas the center line average roughness was 2.4  $\mu\text{m}$  for Comparative Example 5 and 1.6  $\mu\text{m}$  for Comparative Example 6.

In addition, the total amount of contaminants depositing on a surface to be eroded, that is, metal elements with a high melting point other than the major component and alloy components (W, Ti, Ta, Mo, Nb, and the like) and Si, Al, Co, Ni, and B, was 500 ppm or less, that is, in a range of 45 ppm and 360 ppm for Examples 15-20, whereas the total amounts were 60 ppm and 370 ppm for Comparative Examples 5 and 6, respectively.

The hydrogen content of the surface ranged from 1 ppm to 20 ppm in Examples 15-20, whereas the contents were 2 ppm and 20 ppm for Comparative Examples 5 and 6, respectively.

The thickness of the surface damage layer of the surface was 4-20  $\mu\text{m}$  for Examples 15-20, while it was 35  $\mu\text{m}$  for Comparative Example 5 and 25  $\mu\text{m}$  for Comparative Example 6, which had relatively thick surface damage layers.

As shown from a comparison between Tables 8 and 9, the number of nodules and average particle number were small and standard deviation of sheet resistance in a wafer was small for Examples 15-20 according to the present invention, in which none of center line average roughness; total amount of contaminants depositing on the surface to be eroded, that is, metal elements at a high melting point other than the major component and alloy components (W, Ti, Ta, Mo, Nb, and the like) and Si, Al, Co, Ni, and B; hydrogen content; or thickness of the surface damage layer of the surface exceeded the respective upper limits, indicating that good sputtering targets were obtained.

However, in Examples 15 and 18 in which the surface roughness was relatively large and the amount of contaminants was high, and in Example 20 for which the surface roughness was relatively large and the thickness of the surface damage layer was large, the number of nodules and average particle number tended to relatively increase and the standard deviation of sheet resistance in a wafer was slightly higher.

Although Examples 15, 18, and 20 are included in the scope of the present invention and had no particular problems, it is shown that these increases affect the properties of sputtering targets.

In Examples 17-19, in which diamond-finishing machining was performed, the center line average roughness (Ra) was 0.03-0.11  $\mu\text{m}$  and the thickness of the surface damage layer was 4-10  $\mu\text{m}$  and thus the center line average roughness (Ra) and the thickness of the surface damage layer were remarkably small.

In addition, in this case, the number of nodules produced and average particle number were small and the standard deviation of sheet resistance in a wafer at the time of cumulative input power after initiation of use being 10 kWh was constantly low, 2.3-2.8%.

From above, diamond-finishing machining is found to exert a quite excellent effect as in Examples 5-7.

For Examples 18-19, as shown in Table 7, in addition to diamond-finishing machining, wet polishing and washing or chemical polishing, washing, and dehydrogenation treatment were conducted. The number of nodules and average particle number, and the standard deviation of sheet resistance in a wafer were further improved, showing excellent results.

On the contrary, in Comparative Examples 5 and 6, as shown in Tables 8 and 9, the center line average roughness (Ra) was large and the surface damage layer was thick.

The numbers of nodules were large, 17 and 20/target, and the average numbers of appearing particles were also large, 6 and 8/wafer. Especially, the standard deviations of sheet resistance in a wafer were as high as 3.6% and 4.1%, showing clearly bad results.

From above, a copper target of the present invention is shown to be an excellent target with lower incidences of nodule production and appearance of particles, and a small standard deviation of sheet resistance in a wafer. (Examples 21-26 and Comparative Examples 7-8)

Next, examples of application of the present invention to aluminum (Examples 21-26) and Comparative Examples 7-8 are shown.

Sputtering target material composed of highly purified aluminum (Al) was subjected to lathe machining. Then, a surface to be eroded was subjected to diamond-finishing machining, wet polishing, chemical polishing, washing with



ultra-pure water, and dehydrogenation treatment to prepare a sputtering target (300 mm in diameter and 6.35 mm in thickness) with controlled surface roughness (Ra), total amount of contaminants, hydrogen content, and thickness of a surface damage layer. These examples are shown in Tables 10 and 11. In Table 10, a round mark means that a processing or treatment was conducted.

The target thus obtained was connected to a copper backing plate with a diameter of 348 mm and a thickness of 21.0 mm and then subjected to sputtering below.

[Table 10]

[Table 11]

The Al sputtering target thus prepared was set in a DC magnetron sputtering apparatus and sputtering was carried out under a nitrogen atmosphere to form an Al film on a silicon wafer. The number of nodules, average particle number, and standard deviation of sheet resistance ( $\phi 8"$ ) in a wafer at the time of cumulative input power after initiation of use being 10 kWh were examined for Examples 21-26 and Comparative Examples 7 and 8. The results are shown in Table 12.

[Table 12]

As shown in Table 11, the center line average roughness, Ra, was 1.0  $\mu\text{m}$ , the upper limit, or less in Examples 21-26 according to the present invention, whereas it was 3.1  $\mu\text{m}$  for Comparative Example 7 and 2.2  $\mu\text{m}$  for Comparative Example 8.

In addition, the total amount of contaminants depositing on a surface to be eroded, that is, metal elements with a high melting point other than the major component and alloy components (W, Ti, Ta, Mo, Nb, and the like) and Si, Al, Co, Ni, and B, was 500 ppm or less, that is, in a range of 30 ppm and 240 ppm for Examples 21-26, whereas the total amounts were 70 ppm and 400 ppm for Comparative Examples 7 and 8, respectively.

The hydrogen content of the surface ranged from 1 ppm to 20 ppm in Examples 21-26. The contents were 2 ppm and 10 ppm for Comparative Examples 7 and 8, respectively.

The thickness of the surface damage layer of the surface was 5-20  $\mu\text{m}$  for Examples 21-26, while it was 30  $\mu\text{m}$  for Comparative Example 7 and 25  $\mu\text{m}$  for Comparative Example 8, which exhibited thick surface damage layers.

As shown from a comparison between Tables 11 and 12, the number of nodules and average particle number were small and standard deviation of sheet resistance in a wafer was small for Examples 21-26 according to the present invention, in which none of center line average roughness; total amount of contaminants depositing on a surface to be eroded, that is metal elements with a high melting point other than the major component and alloy components (W, Ti, Ta, Mo, Nb, and the like) and Si, Al, Co, Ni, and B; hydrogen content; or thickness of a surface damage layer of the surface exceeded the respective upper limits, indicating that good sputtering targets were obtained.

However, for Examples 21 and 22 in which the surface roughness was relatively high and the amount of contaminants was relatively large among Examples, it is found that the number of nodules was increased and the standard deviation of sheet resistance within a wafer was slightly higher. In Example 26 in which the hydrogen content and the thickness of the surface damage layer were slightly large, the number of nodules and the standard deviation of sheet resistance in a wafer tended to increase, indicating that these increases affect the properties of sputtering targets. However, Examples 21-26 are included in the scope of the present invention and had no problems.

In Examples 23-25 in which diamond-finishing machining was performed, the center line average roughness (Ra) was 0.03-0.15  $\mu\text{m}$ , the amount of contaminants was 30-130 ppm, and the thickness of a surface damage layer was 5-10  $\mu\text{m}$  and thus the center line average roughness (Ra) and the thickness of a surface damage layer were remarkably reduced.

In addition, in this case, the number of nodules produced and average particle number were small and the standard deviation of sheet resistance in a wafer at the time of cumulative input power after initiation of use being 10 kWh was constantly low, 2.4-2.8%.

From above, diamond-finishing machining is found to exert a quite excellent effect as in Examples 5-7.

In Examples 24 and 25, as shown in Table 10, in addition to diamond-finishing machining, wet polishing and washing or chemical polishing, washing, and dehydrogenation treatment were conducted. The number of nodules and average particle number, and the standard deviation of sheet resistance in a wafer were further improved, showing excellent results.

On the contrary, in Comparative Examples 7 and 8, as shown in Table 12, the number of nodules were large, 17-19/target, and the average number of appearing particles were also large, 6 and 12/wafer. In addition, the standard deviations of sheet resistance in a wafer were as high as 4.0% and 3.7%, showing clearly bad results.

From above, an Al target of the present invention is shown to be an excellent target with lower incidences of nodule

production and appearance of particles, and a small standard deviation of sheet resistance in a wafer.

According to the present invention, the surface roughness of a sputtering target is controlled to make the thickness of a thin film formed on a substrate by sputtering even, the amount of contaminants depositing on a surface and the hydrogen content of the surface, and also the thickness of a surface damage layer are reduced so that excellent effects to effectively prevent nodule production and suppress appearance of particles upon sputtering are exerted.

Especially, precision machining using a diamond turning tool can reduce surface roughness effectively without using wet polishing or chemical polishing conventionally required, and selection of this processing condition can reduce the center line average roughness (Ra) to 0.2  $\mu\text{m}$  or less and the thickness of a surface damage layer to 15  $\mu\text{m}$  or less.

By reducing the thickness of the surface damage layer as mentioned above, an excellent effect of shortening a time required for the dummy sputtering remarkably was achieved.

Many widely different embodiments of the present invention may be constructed without departing from the spirit and scope of the present invention. It should be understood that the present invention is not limited to the specific embodiments described in the specification, except as defined in the appended claims.

[Table 1]

	Material	Lathe machining	Diamond-finishing machining	Wet polishing	Chemical polishing	Washing	Dehydrogenation treatment
Example 1	Ti	○		○		○	
Example 2	Ti	○			○	○	○
Example 3	Ti	○		○		○	
Example 4	Ti	○		○		○	
Example 5	Ti	○			○	○	○
Example 6	Ti	○			○	○	○
Example 7	Ti	○	○				
Example 8	Ti	○	○	○		○	
Example 9	Ti	○	○		○	○	○
Comparative Example 1	Ti	○					
Comparative Example 2	Ti	○		○		○	

( A round mark (○) shows that a processing or treatment was conducted. )

[Table 2]

	Surface roughness Ra ( $\mu\text{m}$ )	Total amount of contaminants (ppm)	Hydrogen content (ppm)	Thickness of surface damage layer ( $\mu\text{m}$ )
Example 1	0.8	280	10	40
Example 2	0.6	45	40	10
Example 3	0.9	120	10	10
Example 4	0.3	470	15	10
Example 5	0.2	60	10	8
Example 6	0.08	40	10	6
Example 7	0.17	80	8	11

[Table 2] (continued)

	Surface roughness Ra ( $\mu\text{m}$ )	Total amount of contaminants (ppm)	Hydrogen content (ppm)	Thickness of surface damage layer ( $\mu\text{m}$ )
Example 8	0.13	100	10	7
Example 9	0.07	40	8	5
Comparative Example 1	1.8	670	15	70
Comparative Example 2	3.0	450	10	30

[Table 3]

	Number of nodules (number/target)	Average particle number (number/wafer)	Standard deviation of sheet resistance (%)
Example 1	57	31	3.6
Example 2	23	20	2.4
Example 3	41	26	2.6
Example 4	71	33	3.3
Example 5	30	26	2.4
Example 6	30	26	2.3
Example 7	32	28	2.5
Example 8	31	25	2.3
Example 9	20	21	2.1
Comparative Example 1	500	110	4.8
Comparative Example 2	500	87	3.2

[Table 4]

	Material	Lathe machining	Diamond- finishing machining	Wet polishing	Chemical polishing	Washing	Dehydro- genation treatment
Example 10	Ta	○		○		○	
Example 11	Ta	○			○	○	○
Example 12	Ta	○	○				
Example 13	Ta	○	○	○		○	
Example 14	Ta	○	○		○	○	○
Comparative Example 3	Ta	○					
Comparative Example 4	Ta	○		○		○	

(A round mark (○) shows that a processing or treatment was conducted.)

[Table 5]

	Surface roughness Ra ( $\mu\text{m}$ )	Total amount of contaminants (ppm)	Hydrogen content (ppm)	Thickness of surface damage layer ( $\mu\text{m}$ )
Example 10	0.7	320	10	30
Example 11	0.5	60	10	15
Example 12	0.14	90	10	10
Example 13	0.07	150	10	8
Example 14	0.05	35	8	8
Comparative Example 3	2.2	560	10	55
Comparative Example 4	3.5	480	10	30

[Table 6]

	Number of nodules (number/target)	Average particle number (number/wafer)	Standard deviation of sheet resistance (%)
Example 10	65	33	3.2
Example 11	39	32	2.7
Example 12	28	26	2.6
Example 13	34	29	2.7
Example 14	22	23	2.3
Comparative Example 3	500	95	4.6
Comparative Example 4	500	102	3.3

[Table 7]

	Material	Lathe machining	Diamond- finishing machining	Wet polishing	Chemical polishing	Washing	Dehydrogen- ation treat- ment
Example 15	Cu	○		○		○	
Example 16	Cu	○			○	○	○
Example 17	Cu	○	○				
Example 18	Cu	○	○	○		○	
Example 19	Cu	○	○		○	○	○
Example 20	Cu	○			○	○	○
Comparative Example 5	Cu	○					

[Table 7] (continued)

	Material	Lathe machining	Diamond-finishing machining	Wet polishing	Chemical polishing	Washing	Dehydrogenation treatment
Comparative Example 6	Cu	○		○		○	
(A round mark (○) shows that a processing or treatment was conducted.)							

[Table 8]

	Surface roughness Ra (μm)	Total amount of contaminants (ppm)	Hydrogen content (ppm)	Thickness of surface damage layer (μm)
Example 15	0.8	360	10	10
Example 16	0.4	70	10	10
Example 17	0.11	60	1	4
Example 18	0.05	260	10	10
Example 19	0.03	45	20	6
Example 20	0.9	55	20	20
Comparative Example 5	2.4	60	2	35
Comparative Example 6	1.6	370	20	25

[Table 9]

	Number of nodules (number/target)	Average particle number (number/wafer)	Standard deviation of sheet resistance (%)
Example 15	21	13	2.8
Example 16	8	3	2.6
Example 17	3	1	2.3
Example 18	17	7	2.8
Example 19	6	3	2.4
Example 20	11	5	3.5
Comparative Example 5	17	6	4.1
Comparative Example 6	20	12	3.6

[Table 10]

	Material	Lathe machining	Diamond-finishing machining	Wet polishing	Chemical polishing	Washing	Dehydrogenation treatment
Example 21	Al	○		○		○	
Example 22	Al	○			○	○	○
Example 23	Al	○	○				
Example 24	Al	○	○	○		○	
Example 25	Al	○	○		○	○	○
Example 26	Al	○			○	○	○
Comparative Example 7	Al	○					
Comparative Example 8	Al	○		○		○	

(A round mark (○) shows that a processing or treatment was conducted.)

[Table 11]

	Surface roughness Ra ( $\mu\text{m}$ )	Total amount of contaminants (ppm)	Hydrogen content (ppm)	Thickness of surface damage layer ( $\mu\text{m}$ )
Example 21	0.9	240	10	10
Example 22	0.8	100	10	10
Example 23	0.15	30	1	5
Example 24	0.12	130	10	10
Example 25	0.03	30	15	5
Example 26	0.3	45	20	20
Comparative Example 7	3.1	70	2	30
Comparative Example 8	2.2	400	10	25

[Table 12]

	Number of nodules (number/target)	Average particle number (number/wafer)	Standard deviation of sheet resistance (%)
Example 21	15	7	2.8
Example 22	10	3	2.8
Example 23	2	1	2.4
Example 24	13	6	2.8
Example 25	5	3	2.4
Example 26	10	5	3.5

[Table 12] (continued)

	Number of nodules (number/target)	Average particle number (number/wafer)	Standard deviation of sheet resistance (%)
Comparative Example 7	17	6	4.0
Comparative Example 8	19	12	3.7

## Claims

1. A sputtering target characterized in that the surface roughness of a surface to be eroded of the sputtering target is 1.0  $\mu\text{m}$  or less in terms of center line average roughness (Ra).
2. A sputtering target characterized in that the surface roughness of a surface to be eroded of the sputtering target is 1.0  $\mu\text{m}$  or less in terms of center line average roughness (Ra) and the total amount of contaminants depositing on the surface to be eroded, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B, is 500 ppm or less.
3. A sputtering target according to claim 2, characterized in that the total amount of contaminants, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B, is 300 ppm or less.
4. A sputtering target according to any one of claims 1-3, characterized in that the hydrogen content of a surface to be eroded of the sputtering target is 50 ppm or less.
5. A sputtering target according to claim 4, characterized in that the hydrogen content of the surface to be eroded of the sputtering target is 30 ppm or less.
6. A sputtering target characterized in that the surface roughness of a surface to be eroded of the sputtering target is 1.0  $\mu\text{m}$  or less in terms of center line average roughness (Ra) and the thickness of a surface damage layer of the surface to be eroded is 50  $\mu\text{m}$  or less.
7. A sputtering target characterized in that the surface roughness of a surface to be eroded of the sputtering target is 0.2  $\mu\text{m}$  or less in terms of center line average roughness (Ra) and the thickness of a surface damage layer of the surface to be eroded is 15  $\mu\text{m}$  or less.
8. A sputtering target according to claim 6 or 7, characterized in that the total amount of contaminants depositing on a surface to be eroded, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B, is 500 ppm or less.
9. A sputtering target according to claim 8, characterized in that the total amount of contaminants depositing on a surface to be eroded, metal elements with a high melting point other than the major component and alloy components, and Si, Al, Co, Ni, and B is 300 ppm or less.
10. A sputtering target according to any one of claims 6-9, characterized in that the hydrogen content of a surface to be eroded of the sputtering target is 50 ppm or less.
11. A sputtering target according to any one of claim 6-9, characterized in that the hydrogen content of a surface to be eroded of the sputtering target is 30 ppm or less.
12. A method of manufacturing a sputtering target characterized in that finishing of the sputtering surface is conducted by precision machining using a diamond turning tool.
13. A method of manufacturing a sputtering target characterized in that finishing of the sputtering surface is conducted by precision machining using a diamond turning tool followed by polishing.



European Patent  
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## EUROPEAN SEARCH REPORT

Application Number  
EP 98 10 6493

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	DATABASE WPI Section Ch, Week 9201 Derwent Publications Ltd., London, GB; Class L03, AN 92-003643 XP002071755 & JP 03 257 158 A (TOSHIBA KK)	1,6,7	C23C14/34
Y	* abstract *	2-5,8-11	
Y	ISHIGAMI T ET AL: "HIGH PURITY TI SPUTTER TARGET FOR VLSIS" TOSHIBA REVIEW, no. 161, 1987, pages 38-41, XP002053985 * table 1 *	2-5,8-11	
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A	US 4 663 120 A (PARENT EDWARD D ET AL) 5 May 1987 * column 1, line 37 - line 53 *	12,13	TECHNICAL FIELDS SEARCHED (Int.Cl.6) C23C
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 17 July 1998	Examiner Ekhult, H
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date O : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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